

# PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

### Surface Treated Anodised Aluminium and Aluminium Alloy Articles

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to improvements in or relating to the production of anodised aluminium and aluminium alloy articles surface treated with organopolysiloxanes.

Normally in the production of anodised aluminium and aluminium alloy articles hitherto, it has been the practice to treat the article with boiling water after the anodic coating has been applied electrolytically and the article has been thoroughly deacidified by washing it with cold water, and if desired dyeing it. This has the effect of gradually sealing the absorptive anodic coating as a result of chemical hydration of the aluminium oxide of the coating to aluminium hydroxide, as a result of which the surface coating loses its absorbency. The treatment with boiling water may be effected by the purity of the water and requires careful control to obtain consistent results. Moreover such treatment occupies a substantial period of time which is the longer the greater the thickness of the anodic coating. Surface treatment of anodised aluminium and aluminium alloy articles with organo-polysiloxanes under suitable conditions confers upon them water repellent and other non-sticking properties.

It is known to provide anodised aluminium and aluminium alloy articles with a surface treatment with an organopolysiloxane resin by applying a solution of the resin in a volatile organic solvent to the dried but still unsealed anodised article. For this purpose the treatment with boiling water is omitted,

and the evaporation of water from the deacidified anodised article must be carried out at a temperature insufficient to produce any appreciable chemical hydration.

According to the present invention an anodised unsealed deacidified aluminium or aluminium alloy article is treated with an aqueous emulsion of an organopolysiloxane fluid whereby water is displaced from the pores in the anodic coating which become filled with the organopolysiloxane fluid and thus become sealed.

If the article is to be dyed, this may be done after washing and before the application of the organopolysiloxane fluid and at a temperature insufficient to cause hydration of the anodic coating.

Suitable organopolysiloxane which may be used in our process include, for example trimethylsilyl end-stopped substantially linear polydimethyl siloxanes and similar products in which some of the methyl groups in the linear polydimethyl siloxane are replaced by phenyl or hydroxyl groups or hydrogen; the liquid cyclic polydimethyl siloxanes, for example octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane, all of which are examples of organopolysiloxane fluids.

The amount of organopolysiloxane fluid in the aqueous emulsion applied to the anodised article is preferably sufficient to effect complete sealing of the anodised coating from penetration by water. For a normal anodised film of thickness 0.75—1 mil amounts of the order of less than 0.002 lb./sq. foot are adequate. The effectiveness of sealing may be most readily tested by attempting to dye the article in an aqueous dyestuff solution. If the sealing is complete the anodic coating will remain completely undyed.

Soft anodised aluminium coatings do not possess sufficient inherent mechanical

[Price 4s. 6d.]

strength for employment in cooking utensils where they are liable to be subjected to scouring and scraping, but hard anodised coatings have more inherent mechanical strength. While the invention is applicable to soft anodised articles, it is especially valuable for hard anodised aluminium and aluminium alloy articles, for instance cooking utensils and structural and decorative metal work. In the case of cooking utensils the foodstuff being cooked, for example by frying or baking, does not stick to the surface, and whether or not the utensil is greased and whether or not the foodstuff contains fat the cooked foodstuff is easily removed from it and the utensil is readily cleaned. The water-repellent and non-adhesive properties are accompanied by increased resistance to corrosion on long exposure to the atmosphere, for example in towns where the air is contaminated by corrosive impurities. This is of particular value in the case of structural and decorative work. The invention has the advantage that the sealing of the wet anodised article takes place rapidly after the organopolysiloxane emulsion has been applied and the process lends itself to use in mass production.

The anodised coating into which an organopolysiloxane fluid has been absorbed retains its non-adhesive and water repellent qualities even after long and repeated exposure to cooking temperatures and when ultimately a tendency for foodstuff to stick to the treated anodised surface is noticed the cooking utensil may be reconditioned by a further application of an aqueous emulsion of organopolysiloxane fluid after it has been scoured.

Our invention is further illustrated by the following examples in which all parts and percentages are by weight.

#### EXAMPLE 1

An aluminium plate 3"×3"×18 gauge was anodised in a bath of 22 per cent sulphuric acid at a temperature of 6° C. The current was 2.5 amperes and the anodising time was 20 minutes. After washing in cold running water the plate was sealed by immersion for 5 minutes in an emulsion at 14° C. containing 3 per cent of a tri-

methysilyl end-stopped dimethyl polysiloxane of viscosity 750 c/s at 25° C. The efficiency of sealing was tested by allowing a drop of anthraquinone violet dye to rest on the sealed film for 5 minutes before rinsing and wet brushing. Only very slight absorption of dye was observed.

#### EXAMPLE 2

An aluminium plate 3"×3"×18 gauge was anodised in a bath of 22 per cent sulphuric acid at a temperature of 13° C. The current was 2.5 amperes and the anodising time was 25 minutes. After washing in cold running water the sample was sealed by immersing for 2 minutes in an emulsion at 10° C. containing 1.5 per cent of a trimethylsilyl end-stopped methyl hydrogenopolysiloxane of viscosity 20 c/s at 25° C. and 5 per cent of a trimethylsilyl end-stopped dimethylpolysiloxane of viscosity 750 c/s at 25° C.

The efficiency of sealing was tested by the method described in Example 1 and found to be similar to that obtained in Example 1.

#### WHAT WE CLAIM IS:—

1. A process for the production of aluminium or aluminium alloy articles of improved water-repellency, anti-stick properties and corrosion resistance which comprises treating an anodised unsealed deacidified aluminium or aluminium alloy article with an aqueous emulsion of an organopolysiloxane fluid whereby water is displaced from the pores in the anodic coating which becomes filled with the liquid organopolysiloxane and thus became sealed.

2. A process as claimed in Claim 1 wherein the organo groups in the organopolysiloxane are methyl groups.

3. A process for the production of aluminium or aluminium articles of improved water-repellency, anti-stick properties and corrosion resistance substantially as hereinbefore described and with particular reference to the foregoing examples.

4. Aluminium or aluminium alloy articles whenever produced by a process claimed in any of Claims 1 to 3.

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Agent for the Applicants.

#### PROVISIONAL SPECIFICATION

### Surface Treated Anodised Aluminium and Aluminium Alloy Articles

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare this invention to be described in the following statement:—

The present invention relates to an improvement in the production of anodised

aluminium and aluminium alloy articles surface treated with organo-polysiloxanes. Surface treatment of anodised aluminium and aluminium alloy articles with organopolysiloxanes under suitable conditions confers upon them water repellent and other non-sticking properties.

In the production of anodised aluminium and aluminium alloy articles as hitherto carried out after the anodic coating has been applied electrolytically and the article has been thoroughly deacidified by washing it with cold water, and if desired dyeing it, it is the practice to treat the article with boiling water. This has the effect of gradually sealing the absorptive anodic coating as a result of chemical hydration of the aluminium oxide of the coating to aluminium hydroxide, as a result of which the surface coating loses its absorbency. The treatment with boiling water may be affected by the purity of the water and requires careful control to obtain consistent results. Moreover such treatment occupies a substantial period of time which is the greater the greater the thickness of the anodic coating.

It is known to provide anodised aluminium and aluminium alloy articles with a surface treatment with an organo-polysiloxane resin by applying a solution of the resin in a volatile organic solvent to the dried but still unsealed anodised article. For this purpose the treatment with boiling water is omitted, and the evaporation of water from the deacidified anodised article must be carried out at a temperature insufficient to produce any appreciable chemical hydration.

According to the present invention, after the aluminium or aluminium alloy article has been anodised and freed from electrolyte by washing it with water at a temperature insufficient to cause sealing and while still wet it is treated with an aqueous emulsion of an organo-polysiloxane liquid, as a result of which water is displaced from the pores in the anodic coating which become filled with the liquid organo-polysiloxane and thus become sealed against the absorption of other materials. The liquid organo-polysiloxane may be a fluid that will remain in the liquid condition on subsequent heating or it may be a resin in the still unresinified state, in which latter case the treatment is completed by subjecting the article to baking at raised temperature so as to cause the organo-polysiloxane to assume a resinous condition. It may also be a solution of a resin in a volatile solvent. If the article is to be dyed, this may be done after washing and before the application of the organo-polysiloxane liquid and at a temperature insufficient to cause hydration of the anodic coating.

The term "organo-polysiloxane" as used herein includes trimethylsilyl end-stopped substantially linear polydimethyl siloxanes and similar products in which some of the methyl groups in the linear polydimethyl siloxane are replaced by phenyl or hydroxyl groups or hydrogen; the cyclic polydimethyl siloxanes, for example octamethyl cyclo-tetrasiloxane, hexamethyl cyclo-trisiloxane

and decamethyl cyclo-pentasiloxane, all of which are examples of organo-polysiloxane fluids, in addition to cross linked organo-polysiloxanes that resinify on subsequent heating, for example alkyl polysiloxanes having a ratio of alkyl groups to silicon atoms of approximately 1:1. Methyl polysiloxanes may be used.

The amount of liquid organo-polysiloxane in the aqueous emulsion applied to the anodised article is preferably sufficient to effect complete sealing of the anodised coating from penetration by water. This may be most readily tested by attempting to dye the article in an aqueous dyestuff solution. If the sealing is complete the anodic coating will remain completely undyed.

Soft anodised aluminium coatings are usually very thin and do not possess sufficient inherent mechanical strength for employment in cooking utensils where they are liable to be subjected to scouring and scraping, but hard anodised coatings have more inherent mechanical strength and are often made with a thicker coating. While the invention is applicable to soft anodised articles, it is especially valuable for hard anodised aluminium and aluminium alloy articles, for instance cooking utensils and structural and decorative metal work. In the case of cooking utensils the foodstuff being cooked, for example by frying or baking, does not stick to the surface, and whether or not the utensil is greased and whether or not the foodstuff contains fat the cooked foodstuff is easily removed from it and the utensil is readily cleaned. The water-repellent and non-adhesive properties are accompanied by increased resistance to corrosion on long exposure to the atmosphere, for example in towns where the air is contaminated by corrosive impurities. This is of particular value in the case of structural and decorative work. The invention has the advantage that the sealing of the wet anodised article takes place immediately the organo-polysiloxane emulsion has been applied and the process lends itself to mass production.

The anodised coating into which an organo-polysiloxane fluid has been absorbed retains its non-adhesive and water repellent qualities even after long and repeated exposure to cooking temperatures, and when ultimately a tendency for foodstuff to stick to the treated anodised surface is noticed the cooking utensil may be reconditioned by a further application of an aqueous emulsion of organo-polysiloxane fluid after it has been scoured.

#### EXAMPLE 1.

An aluminium plate 3"×3"×18 gauge was anodised in a bath of 22% sulphuric acid at a temperature of 6° C. The current was 2½ amperes and the anodising time was 20 minutes. After washing in cold running

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water the plate was sealed by immersion for 5 minutes in an emulsion at 14° C. containing 3% of an end-stopped dimethyl polysiloxane.

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## EXAMPLE 2

An aluminium plate 3"×3"×18 gauge was anodised in a bath of 22% sulphuric acid at a temperature of 13° C. The current was 2½ amperes and the anodising time was 25 minutes. After washing in cold running water the sample was sealed by immersing for 2 minutes in an emulsion at 10° C. containing 1½% of a methyl hydrogen polysiloxane and 1½% of an end-stopped dimethyl polysiloxane.

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## EXAMPLE 3

An aluminium plate 3"×3"×16 gauge was anodised in a bath of 17% sulphuric

acid at a temperature of 10° C. The current was 2½ amperes and the anodising time was one hour. After washing in cold running water the plate was sealed by immersing in an emulsion at a temperature of 20° C. containing 3% of a methyl polysiloxane resin capable of resinification by heating.

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On subsequent baking for half an hour at 150 the impregnated polysiloxane became hard and resinous. The efficiency of sealing was tested by allowing a drop of anthraquinone violet dye to rest on the sealed film for 5 minutes before rinsing and wet brushing. Only slight absorption of dye was observed for the three examples quoted above.

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